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[Title of the Invention]

METHOD OF DRYING THERMOPLASTIC NORBORNENE RESIN, THE SUBSTRATE FOR MAGNETIC STORAGE MEDIA USING THE NORBORNENE RESIN, THE MAGNETIC STORAGE MEDIUM USING THE SUBSTARTE, AND THE METHOD OF MANUFACTURING THE MAGNETIC STORAGE MEDIUM

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[Document Title] SPECIFICATION

[Title of the Invention]

METHOD OF DRYING THERMOPLASTIC NORBORNENE RESIN, THE SUBSTRATE FOR MAGNETIC STORAGE MEDIA USING THE THERMOPLASTIC NORBORNENE RESIN, THE MAGNETIC STORAGE MEDIUM USING THE SUBSTRATE, AND THE METHOD OF MANUFACTURING THE MAGNETIC STORAGE MEDIUM

[Claim]

What is claimed is:

1. A method of drying a thermoplastic norbornene resin, the method comprising:

drying the thermoplastic norbornene resin under vacuum or under the ordinary pressure and under vacuum, whereby to remove atmospheric gas components and low-boiling-point organic components contained in the resin.

2. The method according to Claim 1, wherein the drying under the ordinary pressure is conducted at any temperature between 80 and 120℃, and the drying under vacuum is conducted under the degree of vacuum of 20 Pa or lower and at any temperature between 80 and 120℃.

3. The method according to Claim 1 or 2, wherein the thermoplastic norbornene resin contains, after the drying, N_2 of 20 ppm or lower, O_2 of 20 ppm or lower, H_2O of 1 ppm or lower, aliphatic components, that are low-boiling-point organic components, of 20 ppb or lower in total, and aromatic components, that are the other low-boiling-point organic components, of 20 ppb or lower in total.

4. A plastic substrate for magnetic storage media, the substrate being manufactured by injection molding the thermoplastic norbornene resin dried by the method described in any of Claims 1 through 3.

5. The plastic substrate according to Claim 4, wherein the substrate contains in the surface thereof 100 or less rugged portions of 1 μ m x 1 μ m or wider in area.

6. The plastic substrate according to Claim 4 or 5, wherein the straightness Pa in the radial direction of the substrate is 1 μ m or less, the micro waviness of the substrate is 500Å or lower, and the average surface roughness of the substrate is 5Å or lower.

7. A magnetic storage medium comprising:

a substrate,

a magnetic layer above the plastic substrate,

a protection layer on the magnetic layer, and

a lubricant layer on the protection layer,

the substrate being the plastic substrate described in any of Claims 4 through 6.

8. The magnetic storage medium according to Claim 7, wherein the output of a strain gauge is 0.5 g or less at the end of continuous and high-speed head seek tests conducted for 24 hr on the magnetic storage medium rotating at 4500 rpm using a low-flying-height head, the flying height thereof is 1 μ".

9. A method of manufacturing a magnetic storage medium, the method comprising the steps of:

drying a thermoplastic norbornene resin by the method described in any of Claims 1 through 3,

injection-molding the dried thermoplastic norbornene resin, whereby to form a plastic substrate,

forming a magnetic layer above the plastic substrate,

forming a protection layer on the magnetic layer, and

forming a lubricant layer on the protection layer.

[Detailed Description of the Invention]

[0001]

[Field of the Invention]

The present invention relates to a method of drying a thermoplastic norbornene resin used as a raw material for the substrate of a magnetic storage medium mounted on the external memory of a computer and such a magnetic memory for storing digital data. The present invention relates also to a substrate for magnetic storage media containing the thermoplastic norbornene resin dried by the method according to the invention, a magnetic storage medium including the substrate according to the invention and a method of manufacturing the magnetic storage medium (hard disk) using the substrate according to the invention.

[0002]

[Prior Art]

As the capacities of the storage apparatuses that use magnetic storage media become larger, the flying heights of the magnetic heads have been lowered to improve the storage densities. For lowering the flying height of a magnetic head, it is required for the magnetic storage medium to have a very flat and smooth surface, that is to have a very precise surface structure. For example, it is required for the conventional nonmagnetic metal substrate such as an aluminum (Al) substrate to be machined precisely.

[0003]

The conventional nonmagnetic metal substrate and the conventional magnetic storage medium using the conventional nonmagnetic metal substrate are manufactured in the following way.

[0004]

Usually, a blank disk, prepared by rolling a molten metal, heating the rolled metal, annealing the heated metal and machining the annealed metal to have the predetermined dimensions, is used for a nonmagnetic substrate. The blank disk is machined to have a predetermined inner diameter and a

predetermined outer diameter. The flatness and the smoothness of the machined blank disk is improved by lapping. A Ni-P layer of 13 μ m in thickness is plated on the blank disk to improve the surface hardness thereof. The surface of the Ni-P layer is polished until the surface roughness Ra of 10 Å is obtained. Final lapping using diamond slurry is applied to the polished surface of the blank disk. Laser zone textures are formed in the contact start stop (CSS) zone of the thus obtained substrate such that the bump height is, for example, 190Å and the bump density is, for example, 30 x 30. Finally, the substrate is washed meticulously, resulting in a substrate for magnetic storage media.

[0005]

A chromium (Cr) undercoating layer of 500Å in thickness, a Co-14Cr-4Ta magnetic layer of 300Å in thickness and a carbon protection layer of 80 Å in thickness are deposited one after another on the substrate for magnetic storage media by the DC sputtering method. The surface of the as deposited laminate is burnished using a burnishing tape and a fluorine lubricant layer of 20Å in thickness is formed on the burnished surface by dip-coating or by spin-coating, resulting in a magnetic storage medium.

[0006]

The method of manufacturing the conventional substrate for magnetic storage media and the method of manufacturing the conventional magnetic storage medium are becoming more complicated to meet the recent requirements for a higher storage density. Moreover, it is required to manufacture a magnetic storage medium with reduced costs while maintaining high functions. Novel magnetic storage media, that use a plastic substrate, have been proposed to meet these contradictory requirements.

[0007]

The method, that manufactures a plastic substrate by molding and, at the same time, forms the CSS zone thereof with excellent productivity, is advantageous to industrially provide magnetic storage media with low

manufacturing costs.

[0008]

However, the plastic substrates manufactured by injection molding synthetic resin pellets are inferior to the metal substrates and the ceramic substrates such as a glass substrate from the view points of surface flatness and smoothness, since rugged surface portions of several μ m in height difference and micro waviness are caused by molding. The rugged portions and the micro waviness are hazardous for realizing a very flat and smooth surface, that the magnetic storage medium is required to have.

[0009]

The magnetic storage medium formed on the substrate including the rugged portions and the micro waviness is also hazardous for reading out data to the magnetic head and for writing data from the magnetic head. Especially when a low-flying-height head conducts continuous seek at a high speed, the flight thereof is not stabilized and, in the end, head crush is caused. Thus, the conventional plastic substrate impairs the durability of the magnetic storage medium.

[0010]

Polycarbonate resins and poly(methyl methacrylate) resins are used for the material of the plastic substrate for magnetic storage media. In addition to these resins, thermoplastic norbornene resins are used for the material of the plastic substrate for magnetic storage media. The thermoplastic norbornene resin, that exhibits excellent thermal resistance, low hygroscopicity and excellent shape stability, is useful to provide excellent magnetic storage media. However, the problems described above are posed also on the substrate made of the thermoplastic norbornene resin.

[0011]

[Problems to be solved by the Present Invention]

In view of the foregoing, it is a first object of the invention to provide a method of drying a thermoplastic norbornene resin for controlling the

specific gas components therein below certain levels.

[0012]

It is a second object of the invention to provide a very reliable plastic substrate, that contains less rugged portions and low micro waviness due to the use of the thermoplastic norbornene resin, the specific gas components therein are suppressed below certain levels by the method of drying described above.

[0013]

It is a third object of the invention to provide a magnetic storage medium using the plastic substrate described above. More in detail, it is the third object of the invention to provide a magnetic storage medium that has a very flat and smooth surface with less rugged portions and low micro waviness and exhibits excellent durability against continuous and high-speed head seek.

[0014]

It is a fourth object of the invention to provide a method of manufacturing the magnetic storage medium described above.

[0015]

[Means for solving the Problems]

The extensive and intensive studies conducted by the present inventors have revealed that the foregoing objects of the invention are achieved by adjusting the gas components contained in a thermoplastic norbornene resin below certain levels by a specific drying method, and by manufacturing a magnetic storage medium using the dried resin.

[0016]

According to a first aspect of the invention, there is provided a method of drying a thermoplastic norbornene resin, the method including: drying the thermoplastic norbornene resin under vacuum or under the ordinary pressure and under vacuum to remove atmospheric gas components including N_2, O_2 and H_2O and low-boiling-point organic components (organic

impurities) including aliphatic components and aromatic components contained in the resin. The aliphatic components include low-molecular-weight aliphatic hydrocarbons and oxidation products of the resin such as alcohol compounds and carboxylic acid compounds. The aromatic components include the residues of the solvents used for synthesizing the resin.

Advantageously, the drying under the ordinary pressure is conducted at any temperature between 80 and 120℃, and the drying under vacuum is conducted under the degree of vacuum of 20 Pa or lower and at any temperature between 80 and 120℃.

[0017]

Advantageously, the thermoplastic norbornene resin contains, after the drying, N_2 of 20 ppm or lower, O_2 of 20 ppm or lower, H_2O of 1 ppm or lower, aliphatic components, that are low-boiling-point organic components, of 20 ppb or lower in total, and aromatic components, that are the other low-boiling-point organic components, of 20 ppb or lower in total.

[0018]

According to a second aspect of the invention, there is provided a plastic substrate for magnetic storage media manufactured by injection molding the thermoplastic norbornene resin dried by any of the methods described above.

Advantageously, the substrate contains in the surface thereof 100 or less rugged portions of 1 μm x 1 μm or wider in area.

Advantageously, the straightness Pa in the radial direction of the substrate is 1 μm or less, the micro waviness of the substrate is 500Å or lower, and the average surface roughness of the substrate is 5Å or lower.

[0019]

According to a third aspect of the invention, there is provided a magnetic storage medium including: a substrate, a magnetic layer above the plastic substrate, a protection layer on the magnetic layer, and a lubricant layer on the protection layer, the substrate being the plastic substrate described above.

Advantageously, the output of a strain gauge is 0.5 g or less at the end of continuous and high-speed head seek tests conducted for 24 hr on the magnetic storage medium rotating at 4500 rpm using a low-flying-height head, the flying height thereof is 1 μ".

[0020]

According to a fourth aspect of the invention, there is provided a method of manufacturing a magnetic storage medium, the method including the steps of: drying a thermoplastic norbornene resin by the method described above, injection-molding the dried thermoplastic norbornene resin to form a plastic substrate, forming a magnetic layer above the plastic substrate, forming a protection layer on the magnetic layer, and forming a lubricant layer on the protection layer.

[0021]

[Modes for carrying out the Invention]

Now the present invention will be explained in connection with the modes for carrying out the present invention.

[0022]

First, a method of drying a thermoplastic norbornene resin according to a first mode for carrying out the invention will be described.

[0023]

The method of drying a thermoplastic norbornene resin according to the first mode dries the thermoplastic norbornene resin under predetermined conditions to remove gas components contained therein.

[0024]

The thermoplastic resin used according to the invention is neither the polycarbonate resin nor the poly(methyl methacrylate) resin used for the substrates of the magnetic optical disks. The thermoplastic resin used according to the invention is a norbornene resin that exhibits excellent thermal resistance, low hygroscopicity and excellent shape stability due to the rigid molecular structure thereof. The commercial products of the

thermoplastic norbornene resin include APEL supplied from Mitsui Chemicals, Inc. and ZEONEX supplied from Nippon Zeon Co., Ltd.

[0025]

As explained earlier, defects such as rugged surface portions of several μm in height difference and micro waviness are caused more easily on the plastic substrate formed by injection-molding the as supplied thermoplastic norbornene resin than on the metal substrate and the ceramic substrate such as a glass substrate. The defects such as rugged portions and micro waviness roughen the substrate surface.

[0026]

It is considered that the defects such as rugged portions and micro waviness are caused depending on the amounts of gases contained in the norbornene resin pellets. The gases contained in the air are dissolved into the resin pellets while the resin pellets are synthesized. Usually, the resin pellets contain atmospheric gas components such as N_2, O_2, H_2O and CO_2 at the level of from several tens ppm to several %. Since the thermoplastic norbornene resin is nonpolar, H_2O and such molecules that exhibit strong polarity hardly penetrate into the thermoplastic norbornene resin. Therefore, the thermoplastic norbornene resin is preferable due to its hygroscopicity much lower than the hygroscopicity of the other resins. However, even the thermoplastic norbornene resin contains gas components at the level of from several ppm to several hundreds ppm. Usually, the resins inclusive of the thermoplastic norbornene resin contain from several tens ppb to several % of low-boiling-point organic components such as the residue of the solvents for synthesizing the resin and low-molecular-weight aliphatic hydrocarbons. It is considered that the surface defects such as rugged portions and micro waviness are caused depending also on the amounts of the low-boiling-point organic components.

[0027]

In short, it is considered that the atmospheric gases and the low-

boiling-point organic components contained in the thermoplastic norbornene resin cause defects such as rugged portions of several μ·m in height difference and micro waviness in the substrate surface.

[0028]

In this specification, the term "atmospheric gas components" is used to designate the gases such as N_2, O_2 and H_2O contained in the air.

The term "low-boiling-point organic components" is used to designate the organic resin components (aliphatic components) and the aromatic components. The organic resin components include aliphatic hydrocarbons having from 3 to 14 carbon atoms such as raw materials, intermediate products and by-products of the norbornene resin, and oxidation products of the resin yielded by the oxidation of the norbornene resin such as aliphatic alcohol compounds having from 3 to 14 carbon atoms and carboxylic acid compounds having from 3 to 14 carbon atoms. The aromatic components include deteriorated antioxidants yielded by deterioration of the antioxidants added to stabilize the resin and residues of the solvents used for synthesizing the resin.

The atmospheric gas components and the low-boiling-point organic components are referred to collectively as the "gas components".

[0029]

For injection-molding a thermoplastic norbornene resin, the resin is crashed and molten in a heating cylinder with a screw heated at any temperature between 300 and 380℃. The molten resin is injected into the cavity of a molding die. Since the injection molding machine has a closed structure from the heating cylinder to the inlet of the molding die, the gases generated between heating cylinder and the inlet of the molding die have no way to escape. The generated gases are transferred together with the molten resin to the cavity of the molding die. In the cavity, the molten resin flows radially and outwards to fill the cavity. The molten resin is cooled and solidified from the surface side of the cavity. When the generated gas amount

is large, the molten resin flowing radially and outwards generates gases also in the cavity. As a result, the gas generation causes defects such as rugged surface portions of several μ m in height difference. The gas generation obstacles the flow of the molten resin. The obstruction against the flow of the molten resin causes micro waviness in the radial direction of the substrate and increased surface roughness of the substrate.

[0030]

When the resin contains O_2 and H_2O, the norbornene structure of the resin is oxidized easily under the molten state thereof between 300 and 380℃, causing modified low-molecular-weight portions therein. The difference between the fluidity of the modified low-molecular-weight portions and the fluidity of the norbornene resin and the difference between the polarity of the modified low-molecular-weight portions and the polarity of the norbornene resin cause defects.

[0031]

The viscosity of the low-molecular-weight organic impurities contained in the resin is different from the viscosity of the norbornene resin. The viscosity difference causes fluidity difference between the low-molecular-weight organic impurities and the molten resin in the molding die. It is suspected that the low-molecular-weight organic impurities work, due to the fluidity difference, as nuclei, thereon defects are caused.

[0032]

As described above, various low-boiling-point gas components contained in the norbornene resin pose many problems.

Quantitative analysis of the gas components contained in the norbornene resin is conducted to know the gas amounts in the conventional thermoplastic norbornene resin. The results of the quantitative gas analysis conducted on a polyolefin thermoplastic norbornene resin (ZEONEX 280R supplied from Nippon Zeon Co., Ltd.) are listed in Tables 1 and 2.

[0033]

Table 1

Contents of atmospheric gas components in the resin samples (ppm)

	N_2	O_2	H_2O
Lot A	880	355	10.2
Lot B	809	257	7.9
Lot C	623	230	5.1

[0034]

The contents of the atmospheric gas components are analyzed by measuring the amounts of the gases generated by heating a resin pellet of 15 mg from 30 to 300℃ at the heating rate of 10℃/min under the degree of vacuum of 5 x 10^{-9} Torr or lower by the thermal decomposition spectroscopy (TDS).

[0035]

Table 2

Contents of organic impurities (ppb)

	Aliphatic components (C_6-C_{14})		Aromatic components	
	Aliphatic hydrocarbons	Aliphatic alcohol and carboxylic acid compounds	Residues of solvents	Degraded antioxidants
Lot A	100	60	40	20
Lot B	50	30	30	15
Lot C	30	10	20	10

[0036]

The contents of the organic impurities are analyzed by absorbing the gases generated by heating resin pellets of 10 gr. at 120℃ to active carbon, desorbing the absorbed gases from the active carbon by the purge and trap (P & T) method and analyzing the desorbed gases by the gas chromatograph-mass spectroscopy (GC-MS).

[0037]

More in detail, the atmospheric gas components (N_2, O_2 and H_2O) in Table 1 are analyzed in the following way. A resin pellet (15 mg) is heated

from 30 to 300℃ at the heating rate of 10℃/min under the degree of vacuum of 5 x 10^{-9} Torr or lower in a thermal decomposition spectrometer (TDS supplied from ESCO, Ltd.). The gases generated from the heated resin pellet are transferred to a quadrupole mass spectrometer. Nitrogen gas N_2 is detected and measured quantitatively at M/Z = 28, O_2 at M/Z = 32, and H_2O at M/Z = 18.

[0038]

The organic impurities in Table 2 are analyzed in the following way. Resin pellets of 10 gr. are heated at 120℃. The organic gases generated from the heated resin pellets are absorbed to active carbon. The absorbed organic gases are desorbed from the active carbon by the purge and trap (P & T) method. The desorbed organic gases are analyzed qualitatively and quantitatively with a gas chromatograph-mass spectrometer (P & T GC-MS supplied from Shimadzu Corp.).

[0039]

The results described in Tables 1 and 2 indicate the following things.

[0040]

(1) Among the atmospheric gas components, the contents of N_2 and O_2 in the as supplied norbornene resin are high. The content of H_2O in the as supplied norbornene resin is much lower than those of N_2 and O_2. However, H_2O of around 10 ppm accelerates expansion and deformation of the resin when the resin is exposed to a high-temperature atmosphere.

[0041]

(2) The contents of low-molecular-weight aliphatic hydrocarbons, that are the raw materials and the by-products of the norbornene resin, and the contents of aliphatic alcohol compounds and carboxylic acid compounds (oxidation products of the resin) are high. The as supplied norbornene resin also contains aromatic components such as degradation products of the antioxidant yielded by the degradation of the antioxidant added to stabilize the resin and residues of the solvents used for synthesizing the resin.

[0042]

To examine how the gas components described above affect the substrate surface made of norbornene resin, experimental substrates are made of undried norbornene resin and the number of rugged surface portions on the substrate, the micro waviness (Wa) in the radial direction of the experimental substrate and the average surface roughness (Ra) in the radial direction of the experimental substrates are measured. Table 3 lists the results.

[0043]

Table 3

	Numbers of rugged portions (>1 μ m)	Wa (Å)	Ra (Å)
Lot A	1200	1000	13.5
Lot B	900	920	12.5
Lot C	700	870	12.0

[0044]

The number of rugged surface portions is measured under an optical microscope. The micro waviness (Wa) and the surface roughness (Ra) are measured with a noncontact optical surface roughness tester (Chapman Surface Roughness Tester supplied from Chapman Instruments).

[0045]

As Table 3 indicates, 700 or more rugged portions of 1 μ m or more in height difference are caused in the surfaces of the experimental substrates. The observed micro waviness is higher than 850 Å. And, the observed average surface roughness is 12 Å or higher. Errors are caused corresponding to the defective portions in the magnetic storage medium manufactured using the substrate having such defective surface portions. When the micro waviness is high or the surface roughness of the magnetic storage medium is large, the magnetic head fails to float at the start of flight or the head floating is damaged, finally causing head crush.

[0046]

The results of the analyses on the atmospheric gas components and the low-boiling-point organic components and the results of the measurements on the number of rugged portions, the micro waviness and the surface roughness indicate that less rugged surface portions are caused and the micro waviness and the surface roughness are improved in the substrate manufactured by injection-molding the resin that contains less atmospheric gas components and less low-boiling-point organic components. Therefore, it is obvious that the gas components contained in the resin cause rugged portions and micro waviness in the surface of the substrate for magnetic storage media and roughen the substrate surface. In other words, it is considered that when molten resin yields more gases, the molten resin flows in the molding die cavity while yielding gases, causing rugged surface portions of several μ m in height difference, higher micro waviness and higher surface roughness due to the obstructed resin flow.

[0047]

Therefore, it is expected that if the gas components in the thermoplastic norbornene resin are removed, the rugged surface portions, the micro waviness and the surface roughness of the substrate for magnetic storage media will be reduced.

[0048]

It is also expected also that if a substrate made of a thermoplastic norbornene resin containing less gas components is used to manufacture a magnetic storage medium including a laminate for magnetic data storage on the substrate, the magnetic storage medium will be provided with a very flat and smooth surface having extremely few micro defects, reduced waviness and reduced surface roughness. The magnetic storage medium having a very flat and smooth surface, that facilitates excellent head flight, will be reliable and durable.

[0049]

Therefore, it is desirable to remove the gas components from the thermoplastic norbornene resin.

[0050]

Now the method of drying a thermoplastic norbornene resin considering the results of the foregoing examinations will be described.

[0051]

The results of the foregoing analyses indicate that the gas components, that cause rugged portions, micro waviness and a rough surface, include atmospheric gas components such as N_2 and O_2 that are gases at the room temperature and organic components having 6 or more carbon atoms that are liquids at the room temperature. Thus, the boiling points of the gas components that should be removed from the thermoplastic norbornene resin distribute in a wide temperature range. The pellet of the thermoplastic norbornene resin is 1.5 mm or more in thickness. Therefore, for removing the gas components not only form the surface portions but also from the central portions of the resin pellets, it is considered that it is effective to dry the resin pellets not only under the ordinary pressure but also under vacuum.

[0052]

The present inventors have found that the gas components contained in the thermoplastic norbornene resin are removed effectively by drying the resin solely under vacuum or under the ordinary pressure and under vacuum.

[0053]

Therefore, the method of drying the resin according to the invention dries the thermoplastic norbornene resin solely under vacuum or under the ordinary pressure and under vacuum. The thermoplastic norbornene resin is dried under the degree of vacuum of 20 Pa or lower at any temperature between 80 and 120℃ for any period between 2 and 16 hr. For drying the thermoplastic norbornene resin under the ordinary pressure, the resin is dried at any temperature between 80 and 120℃ for any period between 12

and 24 hr. Although it is preferable to dry the resin under the ordinary pressure and under vacuum, drying the resin solely under vacuum poses no problem. For drying the resin under the ordinary pressure, the resin may be dried in the air or in a N_2 atmosphere.

[0054]

Although the thermoplastic norbornene resin may be shaped with any shape as far as the shape is favorable for drying the resin, it is preferable to shape the resin with pellets.

[0055]

Now the method of drying the thermoplastic norbornene resin according to the invention will be described.

[0056]

According to the invention, the thermoplastic norbornene resin is dried solely under vacuum or under the ordinary pressure and under vacuum.

[0057]

For drying the thermoplastic norbornene resin solely under vacuum, it is preferable to dry the resin at 80℃ for 16 hr or longer, at 100℃ for 8 hr or longer, or at 120℃ for 4 hr or longer. For drying the thermoplastic norbornene resin under the ordinary pressure and under vacuum it is preferable to dry the resin under the ordinary pressure at 80℃ for 24 hr and under vacuum at 100℃ for 8 hr or longer; under the ordinary pressure at 100℃ for 12 hr and under vacuum at 100℃ for 8 hr or longer; under the ordinary pressure at 100℃ for 24 hr and under vacuum at 80℃ for 8 hr or longer; under the ordinary pressure at 100℃ for 24 hr and under vacuum at 100℃ for 4 hr or longer; under the ordinary pressure at 120℃ for 24 hr and under vacuum at 80℃ for 4 hr or longer; under the ordinary pressure at 120℃ for 24 hr and under vacuum at 100℃ for 2 hr or longer; or under the ordinary pressure at 120℃ for 24 hr and under vacuum at 120℃ for any period between 2 and 4 hr. The drying temperature and the drying period may be combined appropriately within the ranges of the temperatures and

the periods described above.

[0058]

After drying the thermoplastic norbornene resin by the method according to the invention, the contents of the atmospheric gas components and the low-boiling-point organic components are below the respective certain levels.

[0059]

In detail, the content of N_2 is preferably 20 ppm or lower, the content of O_2 20 ppm or lower, the contents of H_2O 1 ppm or lower, the total contents of low-molecular-weight aliphatic hydrocarbons and oxidation products of the resin such as alcohol compounds and carboxylic acid compounds 20 ppb or lower, and the total contents of aromatic components 20 ppb or lower.

[0060]

Now a plastic substrate for magnetic storage media according to a second mode for carrying out the invention will be described.

[0061]

The plastic substrate for magnetic storage media according to the second mode for carrying out the invention is made of a thermoplastic norbornene resin.

[0062]

In detail, the thermoplastic norbornene resin used for the plastic substrate according to the second mode is dried by the above described method of drying according to the first mode for carrying out the invention.

[0063]

The substrate for magnetic storage media is manufactured by injection molding according to the invention. The plastic substrate is manufactured by using a molding die, the stamper thereof is fixed to a commercially supplied injection molding machine and by setting the resin temperature, the injection speed and the closing pressure at respective appropriate values. The temperatures of the stationary side and the mobile side of the molding die are set individually. For example, the resin temperature is set at 350℃,

the injection speed at 170 mm/s, and the closing pressure at 70 kg/cm^2. Typically, the temperatures of the stationary side and the mobile side of the molding die are set at 130℃. A substrate of 95 mm in diameter and 1.27 mm in thickness is manufactured under these conditions.

[0064]

The preferable substrate for magnetic storage media according to the invention includes less than 100 surface defects (rugged surface portions) of 1 μ m x 1 μ m or wider in area in the surface thereof. For the preferable substrate, the straightness Pa in the radial direction thereof is 1 μ m or less, the micro waviness Wa 500Å or lower, and the average surface roughness 5 Å or lower.

[0065]

Now a magnetic storage medium according to a third mode for carrying out the invention will be described.

[0066]

The magnetic storage medium according to the third mode employs the substrate according to the second mode for carrying out the invention.

[0067]

In detail, the magnetic storage medium according to the third mode employs the substrate manufactured by injection-molding the thermoplastic norbornene resin dried by the method according to the first mode for carrying out the invention.

[0068]

Fig. 1 is a cross sectional view of the magnetic storage medium according to the invention.

[0069]

Referring now to Fig. 1, the magnetic storage medium according to the invention includes a plastic substrate 1 made by injection-molding the thermoplastic norbornene resin dried by the method according to the first mode for carrying out the invention, an intermediate layer 2 on the plastic

substrate 1, a nonmagnetic layer 3 on the intermediate layer 2, a magnetic layer 4 on the nonmagnetic layer 3, a protection layer 5 on the magnetic layer 4, and a liquid lubricant layer 6 on the protection layer 5. The intermediate layer 2, the nonmagnetic layer 3, the magnetic layer 4, the protection layer 5, and the liquid lubricant layer 6 are made of the respective conventional materials. In detail, the intermediate layer 2 is a metal layer made, for example, of titanium (Ti). The nonmagnetic layer 3 is an undercoating layer made, for example, of chromium (Cr). The magnetic layer 4 is an alloy layer of a cobalt (Co) alloy such as Co-Cr-Pt and Co-Cr-Ta. The protection layer 5 is made of carbon. The liquid lubricant layer 6 is made of a fluorine lubricant such as perfluoropolyether.

[0070]

Although the magnetic storage medium according to the invention has been described with reference to Fig. 1, various modifications are possible considering the use thereof. For example, the intermediate layer 2 may be omitted.

[0071]

The magnetic storage medium is shaped with any shape suitable to the instrument or the apparatus, thereon the magnetic storage medium is mounted. The magnetic storage medium for a hard disk drive (HDD) is shaped with a circular plate (disk).

[0072]

The magnetic storage medium according to the invention exhibits excellent durability and stability for head seek under the high-speed and continuous test using a floating head. For the magnetic storage medium according to the invention, the output of a strain gauge after 24 hour seek is preferably 0.5 g or less.

[0073]

Now a method of manufacturing a magnetic storage medium according to a fourth mode for carrying out the invention will be described.

[0074]

The method of manufacturing a magnetic storage medium according to the fourth mode includes the steps of: drying a thermoplastic norbornene resin, injection-molding the dried norbornene resin to form a plastic substrate, and forming at least a magnetic layer, a protection layer and a liquid lubricant layer one after another on the plastic substrate. In the step of drying, the norbornene resin is dried under vacuum or under the ordinary pressure and under vacuum to efficiently remove gas components contained in the norbornene resin.

[0075]

The norbornene resin is dried as described in connection with the first mode. The plastic substrate is manufactured by injection molding as described in connection with the second mode.

[0076]

In the steps of forming, a laminate that facilitates magnetically storing data is formed on the plastic substrate in the following way. An intermediate layer 2 is formed on a plastic substrate by sputtering. A nonmagnetic undercoating layer 3 is formed on the intermediate layer 2. A magnetic layer 4 is formed on the nonmagnetic layer 3. A protection layer 5 is formed on the magnetic layer 4. Finally, a liquid lubricant layer 6 is formed on the protection layer 5 by coating a lubricant solution prepared by diluting a lubricant with a solvent.

[0077]

Preferably, the nonmagnetic undercoating layer 3 is a Cr layer. Preferably, the magnetic layer 4 is a Co-14Cr-4Ta alloy layer.

[0078]

The Cr nonmagnetic undercoating layer 3 is formed by sputtering. The Co-14Cr-4Ta magnetic alloy layer 4 is formed by sputtering. The carbon protection layer 5 is formed by sputtering. The carbon protection layer 5 mainly contains usual graphite, or the carbon protection layer 5 is a diamond-like-carbon (DLC) layer. Nitrogen may be added to the carbon

protection layer 5. The lubricant layer 6 is formed by dip-coating or by spin-coating. Preferably, the lubricant is perfluoropolyether (ZDOL supplied from Ausimont S.P.A., AM2001 supplied from Ausimont S.P.A., DEMUNUM-SA supplied from Daikin Industries Ltd., or DEMUNUM-SP supplied from Daikin Industries Ltd.).

[0079]

The intermediate layer 2, the nonmagnetic undercoating layer 3, the magnetic layer 4, the protection layer 5 and the lubricant layer 6 are as thick as the respective counterparts in the conventional magnetic storage media.

[0080]

The constitution of the laminate is not limited to that described above. For example, the laminate that lacks the intermediate layer 2 poses no problem on the magnetic storage medium.

[0081]

Now the invention will be described more in detail below in connection with the embodiments thereof.

[0082]

In drying norbornene resin pellets, drying methods, drying temperatures and drying periods of time are changed and examined as listed in Table 4.

[0083]

Table 4

Methods of drying resin pellets

	Methods of drying	Drying under the ordinary pressure (in N2 atmosphere)		Drying under vacuum (20 Pa or lower)	
		Drying temps. (℃)	Drying periods (hr)	Drying temps. (℃)	Drying periods (hr)
Comparative	Non	—	—	—	—
Embodiment 1	Method 1	—	—	80	8
Embodiment 2	Method 2	—	—	80	16
Embodiment 3	Method 3	—	—	100	2
Embodiment 4	Method 4	—	—	100	4
Embodiment 5	Method 5	—	—	100	8
Embodiment 6	Method 6	—	—	120	2
Embodiment 7	Method 7	—	—	120	4
Embodiment 8	Method 8	—	—	140	2
Embodiment 9	Method 9	80	24	100	8
Embodiment 10	Method 10	100	12	100	8
Embodiment 11	Method 11	100	24	—	—
Embodiment 12	Method 12	100	24	80	4
Embodiment 13	Method 13	100	24	80	8
Embodiment 14	Method 14	100	24	100	2
Embodiment 15	Method 15	100	24	100	4
Embodiment 16	Method 16	120	24	—	—
Embodiment 17	Method 17	120	24	80	2
Embodiment 18	Method 18	120	24	80	4
Embodiment 19	Method 19	120	24	100	2
Embodiment 20	Method 20	120	24	100	4
Embodiment 21	Method 21	120	24	120	2
Embodiment 22	Method 22	120	24	120	4
Embodiment 23	Method 23	140	24	—	—

[0084]

Embodiment 1

Thermoplastic norbornene resin pellets of 2 kg are dried by the drying method 1 under the degree of vacuum of 20 Pa or lower. A part of the dried resin pellets are used to measure the gas contents therein for the evaluation described later.

[0085]

A plastic substrate of 95 mm in diameter and 1.27 mm in thickness is

manufactured by using a molding die, the stamper thereof is fixed to a commercially supplied injection molding machine with the maximum injection-molding pressure of 70 t. The resin temperature is set at 350℃, the injection speed at 170 mm／s, the closing pressure at 70 kg／cm², the temperature of the stationary side of the molding die at 130℃ and the temperature of the mobile side of the molding die at 130℃.

[0086]

Embodiments 2 through 8

Resin pellets are dried in the same way as according to the embodiment 1 except that the resin pellets are dried by any of the methods 2 through 8 described in Table 4, and substrates for magnetic storage media are obtained using the resin pellets dried by the methods 2 through 8. In the drying methods 2 through 8, the degree of vacuum is set at 20 Pa or lower.

[0087]

Embodiments 9 through 23

Resin pellets are dried in the same way as according to the embodiment 1 except that the resin pellets are dried by any of the methods 9 through 23 described in Table 4, and substrates for magnetic storage media are obtained using the resin pellets dried by the methods 9 through 23. In drying the resin pellets under the ordinary pressure, the resin pellets are dried in a nitrogen atmosphere. For drying the resin pellets under vacuum, the degree of vacuum is set at 20 Pa or lower.

[0088]

Embodiments 24 through 46

A magnetic storage medium according to any of the embodiments 24 through 46 is obtained in the following way. A Cr undercoating layer of 500 Å in thickness is deposited by sputtering on the substrate prepared according to any of the embodiments 1 through 23, a Co-14Cr-4Ta magnetic layer of 300Å in thickness on the Cr undercoating layer, and a carbon protection layer of 80Å in thickness on the magnetic layer. After depositing

these layers, the surface of the carbon protection layer is burnished with a burnishing tape, and a fluorine lubricant (FOMBLINZ-DOL supplied from Ausimont S.P.A. and such a commercially supplied fluorine lubricant) is spin-coated on the burnished carbon protection layer, resulting in a magnetic storage medium.

[0089]

Comparative example 1

A substrate according to the comparative example 1 is obtained in the same way as the substrate according to the embodiment 1 except that the resin pellets are not dried according to the comparative example 1.

[0090]

Comparative example 2

A magnetic storage medium according to the comparative example 2 is obtained by forming a laminate in the same way as according to the embodiment 24 on the substrate prepared according to the comparative example 1.

[0091]

Evaluations

The contents of gas components in the resin pellets dried according to the embodiments 1 through 23 and comparative example 1 are measure. The number of rugged portions in the surface of the substrates prepared using the resin pellets dried according to the embodiments 1 through 23 and comparative example 1, straightness (Pa) in the radial directions of the respective substrates, micro waviness (Wa) in substrate surfaces, and average surface roughness are measured.

[0092]

The contents of the gas components are measured by the methods described in connection with Tables 1 and 2. The number of the rugged surface portions, the straightness, the micro waviness and the average surface roughness are measured by the methods described in connection with Table 3.

[0093]

The contents of the gas components in the resin pellets are listed in Table 5. The number of the rugged surface portions, the straightness, the micro waviness and the average surface roughness in the surfaces of the substrates manufactured by injection molding are listed in Table 6.

[0094]

Table 5

Contents of the gas components in resin pellets

	Methods of drying	Atmospheric gas components (ppm)			Low-boiling-point organic components (ppb)	
		N_2	O_2	H_2O	Aliphatic hydro-carbons, alcohol and carboxylic acid compounds.	Aromatic components
Comparative	Non	880.0	360.0	10.0	160	60
Embodiment 1	Method 1	48.0	20.0	1.0	30	20
Embodiment 2	Method 2	19.2	13.5	0.8	18	15
Embodiment 3	Method 3	200.0	50.0	2.5	52	43
Embodiment 4	Method 4	45.0	35.0	1.1	30	18
Embodiment 5	Method 5	16.3	10.3	0.7	15	12
Embodiment 6	Method 6	90.0	22.0	1.0	40	32
Embodiment 7	Method 7	18.5	15.0	0.8	15	10
Embodiment 8	Method 8	16.0	9.2	0.6	48	41
Embodiment 9	Method 9	18.0	15.5	0.9	20	18
Embodiment 10	Method 10	17.5	14.0	0.7	18	15
Embodiment 11	Method 11	100.0	35.0	1.6	19	18
Embodiment 12	Method 12	75.0	28.0	1.1	19	17
Embodiment 13	Method 13	19.0	17.5	0.8	15	15
Embodiment 14	Method 14	60.0	20.0	0.9	18	17
Embodiment 15	Method 15	18.5	11.2	0.8	15	13
Embodiment 16	Method 16	65.0	19.5	1.2	17	15
Embodiment 17	Method 17	25.0	15.5	0.85	15	12
Embodiment 18	Method 18	14.0	14.5	0.8	15	10
Embodiment 19	Method 19	14.5	13.8	0.7	13	13
Embodiment 20	Method 20	13.0	12.8	0.7	17	15
Embodiment 21	Method 21	13.0	12.0	0.7	18	16
Embodiment 22	Method 22	12.0	11.5	0.6	25	20
Embodiment 23	Method 23	25.5	17.5	0.9	45	30

[0095]

Table 6

Rugged surface portions, straightness, micro waviness and average surface roughness

	Methods of drying	Counting under an optical microscope	Measurements with a Chapman Surface Roughness Tester		
		Number of rugged portions (>1 μ m)	Straightness (μ m)	Micro waviness Wa (Å)	Roughness (Å)
Comparative 1	Non	1200	1.70	1000	13.5
Embodiment 1	Method 1	250	1.12	680	8.8
Embodiment 2	Method 2	90	0.93	490	4.8
Embodiment 3	Method 3	500	1.40	780	10.0
Embodiment 4	Method 4	200	1.35	650	7.5
Embodiment 5	Method 5	30	0.60	270	2.8
Embodiment 6	Method 6	350	1.05	650	10.2
Embodiment 7	Method 7	50	0.80	320	3.5
Embodiment 8	Method 8	125	0.95	520	5.5
Embodiment 9	Method 9	50	0.70	420	4.2
Embodiment 10	Method 10	55	0.75	420	4.0
Embodiment 11	Method 11	120	0.95	580	6.5
Embodiment 12	Method 12	110	1.00	590	6.2
Embodiment 13	Method 13	40	0.82	280	3.4
Embodiment 14	Method 14	85	0.90	520	6.0
Embodiment 15	Method 15	45	0.85	290	3.8
Embodiment 16	Method 16	95	0.92	520	5.7
Embodiment 17	Method 17	80	0.90	480	5.3
Embodiment 18	Method 18	25	0.60	250	2.5
Embodiment 19	Method 19	20	0.52	210	2.2
Embodiment 20	Method 20	55	0.62	200	2.0
Embodiment 21	Method 21	65	0.60	220	2.0
Embodiment 22	Method 22	120	0.72	250	2.4
Embodiment 23	Method 23	150	0.95	520	4.8

[0096]

As Tables 5 and 6 indicate, the rugged surface portions in the surface of the substrate for magnetic storage media are reduced and, at the same time, the straightness in the radial direction of the substrate, the micro waviness and the surface roughness are improved with reducing gas contents in the resin.

[0097]

By adjusting the content of N_2 at 20 ppm or lower, the content of O_2 at

20 ppm or lower, the content of H_2O at 1 ppm or lower, the total contents of aliphatic components at 20 ppb or lower, and the total contents of aromatic components at 20 ppb or lower, the rugged surface portions are suppressed at 100 or less, the straightness in the radial direction of the substrate at 1.0 μm or less, the micro waviness at 500Å or lower and the surface roughness at 5Å or lower.

[0098]

More in detail, as the results according to the embodiments 2, 5 and 7 indicate, the desired characteristics including the number of the rugged surface portions, the straightness of the substrate surface, the micro waviness and the surface roughness are obtained by drying the resin under vacuum at 80℃ for 16 hr, at 100℃ for 8 h, or at 120℃ for 4 hr.

[0099]

As the results according to the embodiment 9 indicates, the desired characteristics including the number of the rugged surface portions, the straightness of the substrate surface, the micro waviness and the surface roughness are obtained by drying the resin under the ordinary pressure at 80℃ for 24 hr and under vacuum at 100℃ for 8 hr. As the results according to the embodiments 13 and 15 indicate, the desired characteristics including the number of the rugged surface portions, the straightness of the substrate surface, the micro waviness and the surface roughness are obtained by drying the resin under the ordinary pressure at 100℃ for 24 hr and under vacuum at 80℃ for 8 hr or at 100℃ for 4 hr. As the results according to the embodiments 18, 19 and 21 indicate, the desired characteristics including the number of the rugged surface portions, the straightness of the substrate surface, the micro waviness and the surface roughness are obtained by drying the resin under the ordinary pressure at 120℃ for 24 hr and under vacuum at 80℃ for 4 hr, at 100℃ for 2 hr or at 120℃ for 2 hr.

[0100]

The thermoplastic norbornene resin is dried more effectively by combining the drying under the ordinary pressure and the drying under vacuum. In other words, the gas components staying deep in the resin pellets are removed more effectively by combining the drying under the ordinary pressure and the drying under vacuum.

[0101]

As the results for the embodiments 8, 22 and 23 indicate, although the atmospheric gas components are reduced more by drying at a too high temperature for a too long time, the drying at a too high temperature for a too long time causes thermal decomposition of the resin, increase of the low-boiling-point organic components and, especially, increase of the rugged surface portions.

[0102]

Continuous and high-speed hear seek durability tests for a low-flying-height head are conducted on the magnetic storage medium according to the embodiments 24 through 46 and the comparative example 2. The continuous and high-speed hear seek durability tests are conducted on the basis of the finding that the durability of the magnetic storage medium and the stable flight of the magnetic head are closely related with each other. The continuous and high-speed hear seek durability tests utilize the fact that the output of a strain gauge moving with the magnetic head is changed by the contact of the magnetic head and the rugged portions in the disk surface. The continuous and high-speed hear seek durability tests measure the outputs of the strain gauge to determine the durability of the magnetic storage media.

In detail, the continuous and high-speed head seek durability tests read the outputs (g) of a strain gauge as the indications of the resistance against friction caused in a high-temperature and high-humidity environment between a giant magneto-resistive (GMR) head (for the flying height of 1.0 μ" and for reading out the data stored at the bit density of 10 Gbit/in^2) and a magnetic storage disk. The GMR head conducts a high-speed head seek

continuously for 24 hr at the output frequency of 40.0 Hz within the radial range between 19.0 mm and 44.5 mm of the magnetic storage disk rotating at 4500 rpm. The tests are conducted using a commercially supplied continuous start stop (CSS) tester (supplied from Center for Tribology Inc.). The results are listed in Table 7.

[0103]

Table 7

Results of head seek tests on magnetic storage media

	Methods of drying	Outputs of strain gauge at the end of the seek test (g)
Comparative 2	Non	*
Embodiment 24	Method 1	*
Embodiment 25	Method 2	0.48
Embodiment 26	Method 3	*
Embodiment 27	Method 4	*
Embodiment 28	Method 5	0.25
Embodiment 29	Method 6	*
Embodiment 30	Method 7	0.33
Embodiment 31	Method 8	1.20
Embodiment 32	Method 9	0.46
Embodiment 33	Method 10	0.44
Embodiment 34	Method 11	1.10
Embodiment 35	Method 12	0.85
Embodiment 36	Method 13	0.30
Embodiment 37	Method 14	0.60
Embodiment 38	Method 15	0.50
Embodiment 39	Method 16	0.62
Embodiment 40	Method 17	0.56
Embodiment 41	Method 18	0.20
Embodiment 42	Method 19	0.20
Embodiment 43	Method 20	0.28
Embodiment 44	Method 21	0.43
Embodiment 45	Method 22	0.65
Embodiment 46	Method 23	*

* Impossible to measure since the head fails to float.

[0104]

As Tables 6 and 7 indicate, the magnetic storage medium including any of the substrates, the rugged surface portions, the straightness in the radial directions, the micro waviness and the surface roughness thereof are improved, exhibits excellent head seek durability. In detail, the magnetic

storage medium including the substrate with 100 or less rugged surface portions, the straightness in the radial direction thereof of 1.0 μ m or less, the micro waviness of 500Å or lower and the surface roughness of 5Å or lower, exhibits excellent continuous head seek durability, for that the output of the strain gauge is 0.5 g or less after the 24 hr continuous head seek. The head and the disk with few rugged surface portions, improved straightness in the radial directions, improved micro waviness and improved surface roughness contact with each other less frequently in the low-flying-height, continuous and high-speed head seek tests and facilitate excellent head flight.

[0105]

Thus, the magnetic storage medium according to the invention exhibits excellent durability and reliability in continuous and high-speed head seek tests.

[0106]

[Effect of the Invention]

As described above, the atmospheric gas components (such as N_2, O_2 and H_2O), and the low-boiling-point organic components (including deterioration products and oxidation products of the norbornene resin such as aliphatic hydrocarbons, alcohol compounds and carboxylic acid compounds and aromatic components such as deteriorated antioxidants and residues of the solvents for synthesizing the resin) contained in the thermoplastic norbornene resin are suppressed below certain levels.

[0107]

The substrate for magnetic storage media according to the invention, manufactured by injection-molding the thermoplastic norbornene resin dried by the method according to the invention, has a precisely machined surface that contains few rugged surface portions and exhibits improved straightness in the radial direction of the substrate, improved micro waviness and improved surface roughness.

[0108]

The magnetic storage medium according to the invention, that facilitates stable head flight in the low-flying-height, continuous and high-speed durability tests, is very precise and very reliable.

[0109]

The manufacturing method according to the invention facilitates manufacturing very precise and very reliable magnetic storage media with excellent mass-productivity and with low manufacturing costs.

[Brief Description of the Drawing Figures]

Fig. 1 is a cross sectional view of a magnetic storage medium according to the invention.

[Assignment of the Reference Numerals and Symbols]

1: Plastic substrate made of thermoplastic norbornene resin

2: Intermediate layer

3: Cr undercoating layer

4: Magnetic layer

5: Protection layer

6: Lubricant layer

[Document Title] ABSTRACT

[Abstract]

[Object]

To provide a method of drying a thermoplastic norbornene resin for controlling the specific gas components therein below certain levels, to provide a reliable plastic substrate made of the thermoplastic norbornene resin dried by the method described above, to provide a magnetic storage medium that employs the plastic substrate described above, and to provide a method of manufacturing the magnetic storage medium described above.

[Configuration]

The gas components in thermoplastic norbornene resin pellets are suppressed below certain levels by drying the resin under vacuum or under the ordinary pressure and under vacuum. A substrate for magnetic storage media is manufactured by injection-molding the resin dried by the method according to the invention. The magnetic storage medium, that employs the substrate manufactured according to the invention and includes at least a magnetic layer, a protection layer on the magnetic layer and a liquid lubricant layer on the protection layer, is manufactured by forming these layers one after another on the substrate according to the invention.

[Selected Drawing] Fig. 1

~~【書類名】~~ ~~図面~~ Document Type: Drawing

~~【図1】~~ Fig. 1